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RECENTLY PUBLISHED RESEARCH OF THE
CHEMICAL INSTITUTE OF THE ARMENIAN AFFILIATE OF THE
ACADEMY OF SCIENCES

"Acid Hydrolysis of 2-chloro-2-hepten-6-one," G. T. Tatevosyan, M. I. Melikyan, M. G. Tuteryan, Chem Inst, Acad Sci Armenian SSR, Yerevan

"Doklady Akademii Nauk Armyanskoy SSR" Vol 2, No 1, 1945, pp 9-13

2-Chloro-2-hepten-6-one was treated with cooling with 84.5% H₂SO₄; after standing for 2 days mixture was diluted and neutralized. After extraction with Et₂O, extract was distilled to yield 76.36% 1-methyl-1-cyclohexen-3-one and its semicarbazone.

"New Method of Preparation of the Nitrile of Valeric Acid," M. T. Dangyan and M. A. Oganisyan, Chem Inst, Armenian Acad Sci, Yerevan

"Doklady Akademii Nauk Armyanskoy SSR" Vol 2, No 2, 1945, pp 41-2

Dicyandiamide and valeric acid were mixed, subjected to slow distillation to yield valeronitrile. Some unidentified crystalline solid also obtained.

"New Method of Preparation of the Anide of Heptadecanoic Acid," M. T. Dangyan and M. A. Oganisyan, Chem Inst, Acad Sci Armenian SSR, Yerevan

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"Doklady Akademii Nauk Armyanskoy SSR" Vol 2, No 3, 1945, pp 71-2

Heptadecanoic acid and dicyandiamide were heated to 210-150 for 6 hours. Distillation at 168 mm gave 71% heptadecanamide

"Preparation of Isocaproamide," M. T. Danyan, R. A. Megroyan, G. A. Musakhanyan, Chem Inst, Acad Sci Armenian SSR, Yerevan

"Doklady Akademii Nauk Armyanskoy SSR" Vol 2, No 4, 1945, pp 107-8

Dicyandiamide and isocaproic acid heated to 200-80 for 6 hours to yield 83.3% isocaproamide.

"Acid Saponification of Butyl(3-chloroacetyl)acetic Acid," G. T. Tatevosyan and M. A. Mikogosyan, Chem Inst, Acad Sci Armenian SSR

"Doklady Akademii Nauk Armyanskoy SSR" Vol 3, 1945 pp 15-19

To a solution of $\text{NaC}_2\text{H}_5\text{COO}$, prepared from Na and $\text{HO}_2\text{C}_2\text{H}_5$, in absolute EtOH, there was added with cooling and stirring 2,4-dichloro-2-butene, after which the mixture was boiled for several hours. After distillation of EtOH and acidification by HCl organic layer was distilled to yield di-Et butyl(3-chloroacetyl) malonate. Hydrolysis by refluxing with alcoholic NaOH gave butyl(3-chloroacetyl)-malonic acid. Heating this over free flame, followed by distillation in vacuo, gave butyl(3-chloroacetyl)acetic acid; this was treated with cooling with concentrated H_2SO_4 , allowed to stand at room temperature to yield α -butyl- γ -acetylbutyric acid.

"Dehydration of Methylolobutylcarbinol," G. T. Tatevosyan and A. G. Terkyan, Chem Inst, Acad Sci Armenian SSR

"Izvestiya Armyanskogo Filiala Akademii Nauk SSSR" No 1/2, 1944, pp 73-8

Methylolobutylcarbinol was heated with crystalline CO_2H , with slow distillation to yield 1-methylolobutanol, which was identified by IR, oxidation.

"The Reactions of Derivatives of Thiourea of the ARNOLD Type With Acids," M. T. Danyan, Chem Inst, Acad Sci Armenian SSR

"Izvestiya Armyanskogo Filiala Akademii Nauk SSSR" No 4, 1944, pp 3-18

Syn-Diphenylthiourea (I) and HCO_2H yielded PhNH_2 ; see

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of I and formic acid gave formanilide contaminated with a little PhNH₂. I and AcOH gave AcNHPh. Similarly, EtCO₂H gave propionanilide; PrCO₂H gave butyranilide; BuCO₂H gave valerianilide; BzOH gave BzNHPh; (CH₃CO₂H)₂ gave succinil; o-C₆H₄(CO₂H)₂ gave phthalanil. The corresponding toluid yields are given using sym-di-o-tolylthiourea and sym-Di-p-tolylthiourea.

"Action of Hydrobromic Acid on Methylolobutylcarbinol," O. T. Tatevosyan and M. I. Melikyan, Chem Inst, Acad Sci Armenian SSR

"Izvestiya Armanakogo Filiala Akademii Nauk SSSR" No 5/6, 1944, pp 23-8

Methylolobutylcarbinol was treated at 0° with HBr; after standing for 1 day mixture was gently boiled to yield 1-methyl-1-bromocyclopentane (I). I when heated with quinoline to 190-200° gave 1-methylcyclopentene which was identified by oxidation with KMnO₄.

"Oxidation of 2,4-dichloro-2-butenes. I. Preparation of Oxalic Acid by Oxidation of 2,4-dichloro-2-butenes by Aqueous Solutions of Nitric Acid and of Calcium Nitrate," V. I. Isagulyants and G. M. Meryan, Chem Inst, Acad Sci Armenian SSR

"Izvestiya Armanakogo Filiala Akademii Nauk SSSR" No 5/6, 1944, pp 17-21

2,4-Dichloro-2-butenes and 20.2% HNO₃ were heated to 60° with stirring for 1-2 hours. Evaporation of the solution gave (CO₂H)₂·2H₂O. Oxidation with 30% Os(NO₃)₂ solution gave so low a yield that it is not recommended as a method of preparation.

"Bis(3-chlorocrotyl)barbituric Acid," O. T. Tatevosyan and M. G. Tetoryan, Chem Inst, Acad Sci Armenian SSR

"Izvestiya Armanakogo Filiala Akademii Nauk SSSR" No 5/6, 1944, pp 29-35

To Na in absolute EtOH was added CH₂(CO₂Et)₂, solidified mixture was treated with 2,4-dichloro-2-butenes with cooling and stirring. After heating on a steam bath, the mixture was cooled and treated with NaOEt, followed by 2,4-dichloro-2-butenes. The mixture was again heated to yield, after distillation of EtOH and treatment with acidified water, di-Et bis(3-chlorocrotyl)malonate. Urea in absolute EtOH was treated with above ester, then added to Na in absolute EtOH, and the mixture heated, followed by refluxing. On cooling, urea was added and mixture was boiled, EtOH was distilled off and residue was treated with water and HCl; after standing overnight bis(3-chlorocrotyl)barbituric acid was filtered off. Hydrolysis of either barbituric acid or malonic ester by NaOH gave bis(3-chlorocrotyl)-malonic acid. By heating latter over a free flame,

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followed by distillation in vacuo, bis(3-chlorocrotyl)-acetic acid was obtained. Bis(chlorocrotyl)barbituric acid is very toxic and has no soporific effect.

"Cleavage of Hydrogen Halide From Polyhalogen Derivatives of Hydrocarbons by Liquid Ammonia. II. Cleavage of Hydrogen Halide From Mixed Polyhalide-Substituted Ethanes," G. M. Mkryan, Chem Inst, Acad Sci Armenian SSR

"Izvestiya Armysanskogo Filiala Akademii Nauk SSSR" No 5/6, 1944, pp 45-50

1,2-Dichloro-1,2-dibromoethane, prepared by bromination of $(CH_2Cl)_2$, was cooled to -40° and treated with an excess of liquid NH_3 ; mixture was allowed to warm to -30° (under 300-400 mm pressure) in course of 1.5-2 hours, after which NH_3 was evaporated spontaneously. The organic layer was distilled to yield pure 1,2-dichloro-1-bromoethane. Bromination of this in daylight at room temperature gave 1,2-dichloro-1,1,2-tribromoethane. 1,1,2-Trichloro-1,2-dibromoethane treated with NH_3 as above yielded trichlorobromoethane. 1,2-Dichloro-1,1,2-tribromoethane animated as above in presence of dry Et_2O yielded 1,2-dichloro-1,2-dibromoethane.

"Synthesis of Aniline and Other Amines From Calcium Cyanamide," M. T. Dangyan, Chem Inst, Acad Sci Armenian

"Izvestiya Armysanskogo Filiala Akademii Nauk SSSR" No 1/2, 1942, pp 183

Ca cyanamide was transformed into dicyandiamide, which reacted with $NaOH$ to yield $PhCN$ or $PhNH_2$, latter being readily transformed to $PhNH_2$, with an over-all yield of 70%. No quantitative data are given in this preliminary communication.

"Reaction of Halogens, Aluminum, and Magnesium With Alcohols and Esters," M. T. Dangyan, Chem Inst, Acad Sci Armenian SSR

"Izvestiya Armysanskogo Filiala Akademii Nauk, SSSR" No 3/4, 1942, pp 63-71

Al and $NaOH$ were treated dropwise with Br; after spontaneous reaction subsided, mixture was distilled to yield $MeBr$; similarly powdered Al, $EtOH$, Br gave BuBr; Al, iso-AmOH, Br gave iso-AmBr; Al, octyl alcohol, Br gave, after heating at $80-150^\circ$, octyl bromide; Al, iso-PrOH, iodine gave iso-PrI; iodine, powdered Mg, iso-PrOH gave iso-PrI. Iodine, powdered Mg, (iso-AmO) $_2$ OH, heated carefully until reaction subsided, yielded iso-AmI.

"Reactions of Iodine and Esters With Manganese and Zinc," M. T. Dangyan, Chem Inst, Acad Sci Armenian SSR

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"Izvestiya Armysanskogo Filiala Akademii Nauk SSSR" No 7, 1942, pp 7-14

Iodine, powdered Mn, and BzOMe gave, on slow distillation 33.4% MeI , distillation residue being essentially pure $(\text{BzO})_2\text{Mn}$. Iodine, powdered Zn, BzOMe gave slightly more MeI . Similarly BzOEt with Mn and Zn gave 33.68 and 32.82% EtI , respectively. Iso- AmOAc gave analogously 14.7% iso- AmI with Mn and 12.27% with Zn. (Iso- $\text{AmO})_2\text{CH}$ similarly gave 81.6% iso- AmI with Mn and 53.26% with Zn.

"Acid Saponification of 5-chloro-4-hexenoic Acid," G. T. Tatevosyan, M. I. Melikyan, M. G. Tutaryan, Chem Inst, Acad Sci Armenian SSR

"Izvestiya Armysanskogo Filiala Akademii Nauk SSSR" No 7/6, 1944, pp 37-44

To sodio-malonic ester (from Mn and $\text{CH}_2(\text{CH}_2\text{Et})_2$) in absolute EtOH there was added dropwise with cooling 2,4-dichloro-2-butenes, after which mixture was refluxed and was allowed to stand overnight to yield, after distillation of EtOH and acidification with dilute H_2SO_4 , di-Et (3-chlorocrotyl)malonate. To KOH in H_2O was added above ester and mixture was carefully heated until reaction set in. After boiling for a short time, mixture was cooled, acidified with HCl , and extracted with Et_2O . Removal of Et_2O gave (3-chlorocrotyl)-malonic acid. Heating of this over a free flame, followed by distillation in vacuo gave 5-chloro-4-hexenoic acid. Treatment of latter with concentrated H_2SO_4 with cooling gave, after standing for 1 day, 7-acetylbutyric acid hydrate.

"Reaction of Iodine, Aluminum, and Magnesium With Alcohols, Ethers, and Acetal," M. T. Dangyan, Chem Inst, Acad Sci Armenian SSR

"Izvestiya Armysanskogo Filiala Akademii Nauk SSSR" No 6, 1942, pp 41-6

Iodine and Mg or Al react with esters, ethers, alcohols, and acetal to yield alkyl iodides. PrCO_2Et , iodine, and Al powder yield by spontaneous reaction 56% EtI ; use of Mg lowers yield to 44.6%. Iodine and Al treated with acetal with cooling yield 61.4% EtI and AcH . Iodine and powdered Al treated with cooling with iso- AmOH yield after refluxing 57% iso- AmI . Iodine and Al treated with iso- AmOMe with cooling, followed by heating to 100-300°, yield 50.9% iso- AmI ; use of Mg lowered yield to 45.6%. (Iso- $\text{Am})_2\text{O}$, iodine, and Al gave 84.5% iso- AmI .

"Reaction of Bromine With Esters of Nitrous Acid," M. T. Dangyan and S. A. T. Danielyan, Chem Inst, Acad Sci Armenian SSR

"Izvestiya Armysanskogo Filiala Akademii Nauk SSSR" No 8, 1942, pp 23-6

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EtNO_2 was treated with cooling with Br after which mixture was warmed to 67° for 20 minutes with evolution of HBr and N oxides. Distillation gave EtOAc and EtBr (no amounts stated). Similar reaction with BuNO_2 gave BuBr and BuAc , while use of AmNO_2 gave iso-AmBr and iso-Am isovalerate.

"Preparation of Nitrogenous Organic Compounds by Means of Calcium Cyanamide," M. T. Dangyan, Armenian Br, Acad Sci USSR

"Investiya Armanyskogo Filiala Akademii Nauk SSSR" No 9/10, 1942, pp 53-72

A number of amides, amides, imines, and nitriles were prepared with Ca cyanamide as the N carrier; Ca derivative is transformed into diacyandiamide by action of water in conventional manner and this is actual reagent used. Diacyandiamide (I) and NaOH , heated at $120-210^\circ$ in course of 2.5 hours, under an air reflux condenser, yielded, after extraction with Et_2O and EtOH , followed by distillation of extract, a residue at 280° , essentially pure NaONH_2 ; repetition of experiment with 15-hour refluxing period gave PhON , NH_3 , and unidentified insoluble solid. Doubling amount of NaOH gave nearly twice PhON and NaONH_2 , while a 3-molar ratio of NaOH gave 85% of PhON and 13.8% NaONH_2 . Further increase of NaOH ratio lowers yield of PhON . Further reaction with urea, PhON , EtCO_2H , PrCO_2H , iso- BuCO_2H are decreased.

"Derivatives of Hexachloro-3-hexene," A. N. Akopyan, G. M. Mkryan, N. A. Pabiyon, O. B. Garibaldyan, Chem Inst Armenian Acad Sci

"Investiya Armanyskogo Filiala Akademii Nauk SSSR" No 12, 1942, pp 89-94

Hexachloro-3-hexene in 96% EtOH was treated with stirring with Zn dust added in small portions, with cooling to $30-40^\circ$, in course of 2-3 hours. Solutions was diluted with H_2O , filtered and organic layer distilled to yield 3,4-dichloro-1,3,5-hexatriene. On standing at room temperature for 1 hour, product polymerizes to a rubber, which is soluble in CHCl_3 and CCl_4 . On aging, polymer loses its flexibility and solubility. Chlorination yields original substance, while bromination in CCl_4 yields 1,2,5,6-tetrabromo-3,4-dichloro-3-hexene.

"Reactions of Acetal and Iodine With Magnesium, Zinc and Manganese," M. T. Dangyan, Chem Inst, Armenian Acad Sci

"Investiya Armanyskogo Filiala Akademii Nauk SSSR" No 2, 1941, pp 43-7

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Powdered Mg and iodine, treated with cooling with acetal, followed by distillation, yielded EtI after addition of more acetal to distillation residue and a second distillation. Similar use of powdered Zn gave 1.43 g. EtI from 8.34 g. acetal, while powdered Mn gave 5.01 g EtI from 9.418 g. acetal.

"Reaction of Halogens and Certain Metals With Esters of Ortho Acids," M. T. Danyan, Chem Inst, Yerevan

"Izvestiya Argyanskogo Filiala Akademii Nauk SSSR" No 2, 1941, pp 31-5

Slow distillation of a mixture of iodine, powdered Fe and HC(OEt)_2 (I) gave EtI and HCO_2Et (II). Iodine, Mg and I also gave EtI and II. Likewise iodine, Zn and I gave EtI and II; iodine, Mn and I gave EtI and II. Iodine, powdered Al and HC(OEt)_2 , were mixed with cooling and subjected to slow distillation to yield mixed EtI and Et formate. Similarly iodine, Fe and I gave EtI and Et formate. Similarly iodine, Fe and I gave EtI and Et formate, while iodine, Fe and I gave EtI, mixed Et formate, EtOH. Iodine, (iso-AmO) $_2\text{CH}$ (III) and Al gave iso-AmI and impure iso-AmO. Also iodine, Fe and III gave iso-AmI and impure iso-AmOH.

"Reaction of Halogens and Iron With Alcohols, Esters, and Acetal," M. T. Danyan, Chem Inst, Armenian Acad Sci

"Izvestiya Argyanskogo Filiala Akademii Nauk SSSR" No 3/4, 1941, pp 87-95

Iodine, powdered Fe and EtOPh , refluxed for 10 minutes, distilled to yield 90.75% EtI; using iodine in proportion to form FeI, gave 48.6% EtI. Similarly iodine, acetal and Fe (with cooling) gave EtI. Similarly iso-AmOH gave iso-AmI, EtOH gave EtI, BuOH gave EtI, in 36-64% yields. AmOAc gave with Fe $^{++}$ 74.7% iso-AmI, and 41.4% with Fe $^{+}$.

"Gaseous Products of the Action of Zinc Dust on 2,4-dichloro-2-butene," G. T. Tatevosyan and S. A. Vardanyan, Chem Inst, Armenian Acad Sci

"Izvestiya Argyanskogo Filiala Akademii Nauk SSSR" No 8, 1941, pp 75-8

2,4-Dichloro-2-butene in 80% EtOH was treated with Zn dust with cooling and collection of gases; mixture was finally heated to 50-60°. Products were identified as 2-xylene and 1,3-butadiene by means of their bromides.

"Reaction of Bromine and Iron With Alcohols," M. T. Danyan, Chem Inst, Armenian Acad Sci

"Izvestiya Argyanskogo Filiala Akademii Nauk SSSR" No 7, 1941, pp 41-4

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Powdered Fe and BuOH were treated dropwise with Br, with occasional cooling. After refluxing for 1 hour, 46.4% BuBr was obtained. Similarly, 64.65% MeBr, 55.22% AmBr, and 50.8% octyl bromide were obtained from corresponding alcohols. When Fe and Br were taken in proportions to yield FeBr₃, following yields were obtained: MeBr 33.77%, EtBr 13.6% BuBr 30.9%, AmBr 26%, and octyl bromide 23.2%.

"Synthesis and Degradation of Acetylenic Glycols,"
A. Babayan, Chem Inst, Yerevan

"Investiya Argyanskogo Filiala Akademii Nauk SSSR"
No 5/6, 1941, pp 121-45

A satisfactory method for preparation of acetylenic glycols with 70-80% yields was developed. KOH, Et₂O, and Me₂CO were treated with stirring at 13-15° with dry C₂H₂; after standing mixture was hydrolyzed, organic layer neutralized with CO₂, and distilled to yield 76.6% 2,5-dimethyl-3-hexyne-2,5-diol; slow distillation of this in presence of para-Fe C₆H₅SO₃F gave 80% bis(1-methylvinyl)acetylene. KOH, Et₂O, Me₂CO and C₂H₂ gave analogously 64.6% 3,6-dimethyl-4-octyne-3,6-diol, and a small amount of 3-methyl-1-penten-3-ol dehydrated as above gave 83.6% 3,6-dimethyl-2,6-octadien-4-yne. Other reactions and yields are given, and physical properties described.

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